Performance of an infrared microspectrometer at the NSLS

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A facility to perform infrared microspectroscopy is under development at the NSLS of Brookhaven National Laboratory. The high brightness infrared light produced as synchrotron radiation makes a nearly ideal source for microspectroscopy. High quality spectra from 10 μm sized areas can be acquired in less than 1 min. A description of the installation, microspectroscopy performance, and an example application are presented. © *1995 American Institute of Physics.*

I. INTRODUCTION

Infrared microspectroscopy combines the spatial resolution of a microscope with infrared spectral analysis. The technique became practical following the development of rapid scan FTIR (Fourier transform infrared) spectrometers in the 1980's. The primary spectral range is the mid-infrared (wavelengths from 2.5 to 15 μm or 650 to 4000 cm^{-1}), known as the molecular fingerprint region due to the unique absorption features of many chemical species[1]. Material defects, such as found in semiconductor crystals, can also be located and characterized[2].

The demands on the IR source, detector, and optical quality are more rigorous than for standard IR spectroscopy. Sensitive, small area detectors and special optical elements that perform at the diffraction limit are available in existing systems. Like other microscopies, a very bright source of light is needed, yet the standard source for mid-infrared microspectroscopy is the globar; a T~1200K blackbody source of moderate brightness. In comparison, infrared synchrotron radiation (IR-SR) can be 3 orders of magnitude brighter[3]. Two infrared beamlines have been established at the NSLS VUV ring, with one (designated U2B) devoted to high brightness spectroscopy in the mid- and near-IR. Excellent performance has already been demonstrated for other high brightness spectroscopic applications[4]. A description of the beamline and optics are given elsewhere[5].

II. INSTRUMENTATION

The potential for microscopy with IR-SR can be illustrated by ray tracing calculations. The U2B beamline collects 8×12 mrad and delivers an IR flux comparable to the globar source of commercial FTIR spectrometers. When this flux is focused by an ~ f/0.7 (N.A.=0.58) objective at the sample stage of a microscope, ray tracing predicts an illuminated area 10 μm × 20 μm. The shape and size is determined by small aberrations (spherical aberration and coma) introduced by the beamline's spherical mirrors. If the beamline's optical elements were replaced with ideal ellipsoids, the synchrotron source would produce a diffraction-limited spot, i.e. ~2 to 10 μm, for the principal wavelength range of interest. Thus, an additional order of magnitude in brightness might be gained with such custom optics. Still, when compared to the globar's spotsize of >100 μm diameter, one anticipates two orders of magnitude higher brightness using IR-SR with the existing optics at U2B.

Preliminary tests of the IR-SR source for microspectroscopy were conducted with a commercial instrument; a Spectra Tech "IRμs"™ scanning infrared microspectrometer. The IRμs™ consists of a rapid scan FTIR spectrometer and IR microscope integrated into one instrument. Two sets of apertures, located at secondary focuses immediately before and after the primary sample focus, are used to control the size and shape of the illuminated spot at the sample position and also reject scattered or diffracted light. The tests began with transmission measurements through small apertures at the primary sample focus, and immediately confirmed the anticipated superior performance using the infrared synchrotron source. In particular, signal levels through a 13 μm diameter aperture were found comparable to those obtained using the internal globar source through a 100 μm aperture. This confirms a two order-of-magnitude brightness advantage.

The intrinsic (i.e. with no secondary apertures) IR-SR spotsize produced at the IRμs™ sample location was determined by scanning a 3 μm diameter aperture across the focus and measuring the transmitted intensity. At a wavelength of 5 μm, the IRμs™ 15x objective (N.A. = 0.58) produced a FWHM intensity profile approximately 15 μm × 20 μm (see Fig. 1), consistent with the ray tracing prediction. We again note that a factor of two or three smaller spotsize could be realized by eliminating the mirror aberrations (e.g. replacing sphericals with ellipsoids), leading to better confinement of the beam and an additional order of magnitude improvement in brightness.

III. PERFORMANCE

We next show that the brightness advantage translates to higher performance spectroscopy for small specimens.
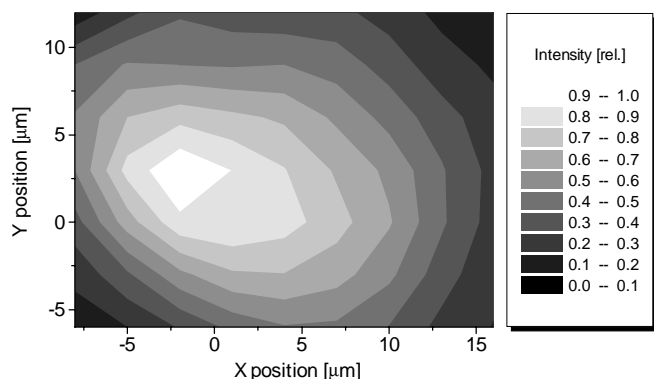
FIG. 1. Intensity profile map of the intrinsic spotsize at the Irµs™ sample focus using the IR-SR source at the U2B beamline. A representative wavelength of 5 µm was chosen.

FIG. 3. Absorbance maps for a CdZnTe single crystal wafer. A 100 µm X 160 µm area was scanned at 10 µm intervals. Left panel: absorbance at $\lambda = 6$ µm: right panel: $\lambda = 2.5$ µm

The available signal-to-noise can be illustrated by ratioing two spectra with no sample. A plot of this ratio (versus frequency or wavelength) produces a line around 100% (i.e. unity), with deviations from the 100% value an indication of noise. Such "100% lines" can be measured through various apertures to indicate the performance available for small
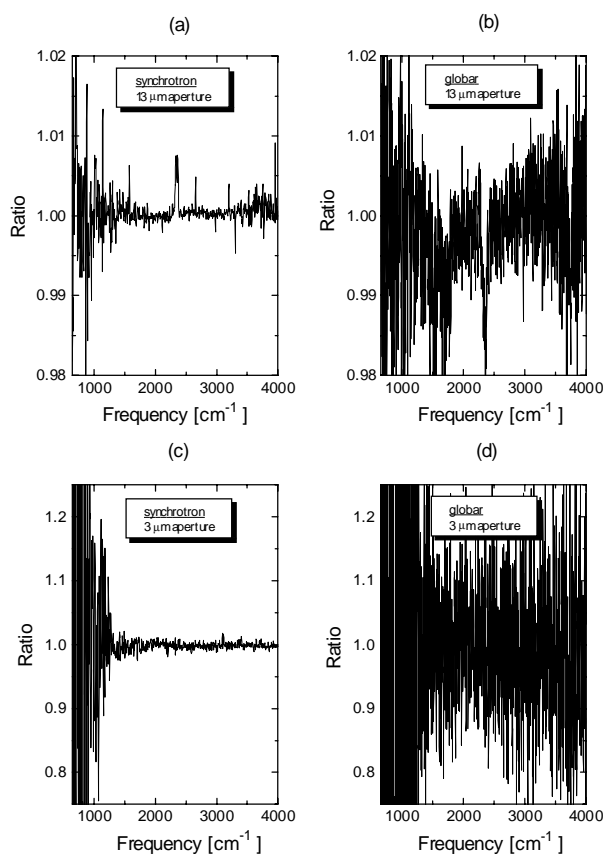

FIG. 2. "100% lines" for small apertures, comparing synchrotron and globar sources. Upper panels compare 13 µm pinhole aperture signal-to-noise for (a) synchrotron source and (b) globar source. Lower panels compare 3 µm pinhole aperture signal-to-noise for (c) synchrotron source and (d) globar source.
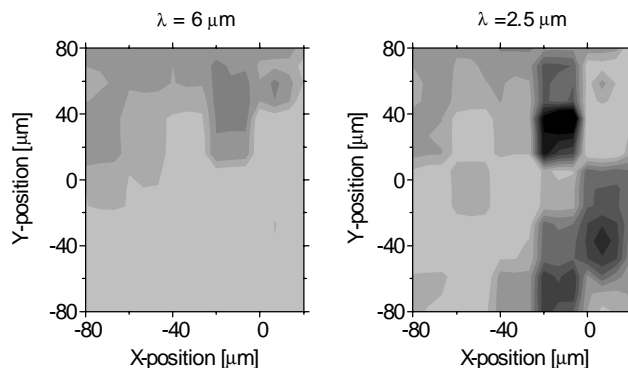
specimens. Figure 2 shows that performance for two different apertures using both the synchrotron and globar (internal) sources. Except for features due to variations in the ambient CO_2 and H_2O air concentrations (e.g. near 2400 cm^{-1}), the noise is predominantly random. The time to scan each spectra was about 1 minute for the 13 µm aperture and 4 minutes for the 3 µm aperture. In both cases the IR-SR source outperforms the globar. This is particularly true for the smaller aperture where the noise for the IR-SR source is more than 20 times smaller. To achieve the same S/N ratio with the globar, one would need to signal average at least 400 times longer, so a four minute measurement with IR-SR would take more than 1 day with the globar. We also note that reasonable performance is achieved through the 3 µm diameter aperture for wavelengths out to 7 µm (1400 cm^{-1}). This demonstrates the potential of high brightness IR-SR for spectroscopy beyond the diffraction limit (e.g. near-field).

One application of spectroscopic mapping is for locating and identifying material defects in semiconductors. For example, oxygen forms a defect in silicon[2] that can be identified by well-defined absorption features near 1100 cm^{-1}. In a similar fashion, we have attempted to characterize the nature of defects in CdZnTe single crystal wafers; a substrate material for many II-VI semiconductor devices. Tellurium frequently forms precipitates a few microns in size, and these may affect the performance of devices grown on the substrate's surface. The absorbance features of Te precipitates are unknown, but using bulk Te as a guide, we expect increasing absorbance for wavelengths less than 3 µm. IRµs™ spectra for individual Te precipitates are consistent with this expectation. Absorbance maps for wavelengths longer (6 µm) and shorter (2.5 µm) than this appear to differentiate regions containing Te. Figure 3 reveals a Te precipitate at the location $x = -10$ µm, $y = +40$ µm. The microscope focus was set to a position 500 µm deep into the sample. The other absorption features near $x = 0$ µm, $y = -40$ µm for $\lambda = 2.5$ µm suggest other Te precipitates, either smaller in size or at a different depth in the CdZnTe wafer.

IV. CONCLUSIONS

A facility has been established on the NSLS VUV ring to perform IR microspectroscopy at spatial resolutions previously unattainable. The very high brightness and low noise of infrared synchrotron radiation enables such performance. Based on the good signal-to-noise for spectra measured through apertures only a few microns across, spatial resolution beyond the diffraction limit appears feasible.

A number of research activities have begun or are being planned. One of the main activities will be locating and identifying defects in semiconductor materials, including infrared devices. The facility will also be used for micro-characterization of various composites and laminates, and both biological and forensic specimens. We are also considering spectrometer modifications to widen the spectral range and incorporate both photo-modulation and time-resolved spectroscopy capabilities.

[1] For example, see "9th International Conference on Fourier Transform Spectroscopy", SPIE conf. proc. vol. 2089 (held August 23-27, 1993 - Calgary, Alberta, Canada).

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[5] G.P. Williams, G.L. Carr, and M. Hanfland, *Rev. Sci. Instr.* **66**, 1643 (1995)..